We claim:

1. A process for preparing acid formates which comprises

(a) partially hydrolyzing methyl formates with water;

(b) separating off by distillation methyl formate and methanol from the reaction mixture obtained in process stage (a), forming a stream comprising formic acid and water;

(c) converting the stream comprising methyl formate with or without methanol from the process stage (b) by

(i) reaction with a basic compound having a pK_a of the conjugate acid of the appropriate dissociation state of ≥ 3, measured at 25°C in aqueous solution, in the presence of water, and

(ii) removal of the methanol by distillation, into a stream comprising formate and water; and

(d) combining the stream comprising formic acid and water from the process stage (b) and the stream comprising formate and water from the process stage (c), forming a mixture comprising the acid formate and water.

2. A process as claimed in claim 1, wherein, in the process stage (a), the methyl formate and the water are fed in a molar ratio of 0.1 to 1.

3. A process as claimed in claims 1 to 2, wherein, in the process stage (c), the removal of the methanol by distillation and the reaction of the methyl formate with the water and basic compound with transfer into the stream comprising formate and water are carried out together in one column.

Drawing

4. A process as claimed in claims 1 to 3, wherein, in the process stage (d)

(i) the stream comprising the formic acid and the water from the process stage (b), together with the mother liquor recirculated from step (iv), is concentrated in a column or an evaporator with removal of water by distillation;

(ii) the stream which was produced from step (i) by concentration and comprises formic acid, water and formate is combined with the stream comprising the formate and water from the process stage (c) forming a mixture comprising the acid formate and water;

(iii) solid acid formate from the mixture comprising acid formate and water obtained from step (ii) is precipitated by crystallization and this is isolated; and

(iv) the resultant mother liquor is recirculated to step (i).

5. A process as claimed in claims 1 to 3, wherein, in process stage (d)

(i) the stream from the process stage (b) comprising the formic acid and the water and the stream from the process stage (c) comprising the formate and the water are combined to form a mixture comprising the acid formate and water in a column or an evaporator with removal of water by distillation; and

(ii) solid acid formate is separated off by spray granulation, spray drying or melt crystallization from the mixture obtained from step (i) comprising acid formate and water, and this solid acid formate is isolated.

6. A process as claimed in claims 1 to 5, wherein, in process step (c), the basic compound is sodium hydroxide, sodium hydrogen carbonate, sodium carbonate, potassium hydroxide, potassium hydrogen carbonate, potassium carbonate and/or ammonia.

7. A process as claimed in claims 1 to 6, wherein the acid formate prepared is acid potassium formate, acid sodium formate, acid calcium formate or mixtures thereof.

8. A process as claimed in claims 1 to 7, wherein the acid formate prepared is potassium diformate, sodium diformate, sodium tetraformate or mixtures thereof.

9. An apparatus for preparing acid formates as claimed in claims 1 to 8, comprising:

(a) a reactor (A) suitable for hydrolyzing methyl formate;

(b) a column (B) suitable for separating by distillation a stream comprising methyl formate, formic acid, methanol and water into methyl formate, methanol and a stream comprising formic acid and water, which column is connected on the feed side to the reactor (A);

(c) a column (C) suitable for saponifying methyl formate with a basic compound and for removing methanol by distillation, which column is connected on the feed side to the column top of column (B) and has above said feed an inlet point for the basic compound; and

(d) a column (D) suitable for removing water from a stream comprising formic acid and water, which column is connected on the feed side to the column bottom of column (B).

10. An apparatus as claimed in claim 9, comprising

(e) an apparatus (E) suitable for crystallizing acid formate, which apparatus is connected on the feed side to the column bottom of column (D) and to the column bottom of column (C);

(f) an apparatus (F) suitable for separating off crystals of the acid formate, which apparatus is connected on the feed side to apparatus (E); and

(g) a connection line (11) between apparatus (F) and column (D), which connection line is suitable for recirculating mother liquor.

11. An apparatus as claimed in claim 9, comprising

(e) a connection line (8) between the column bottom of column (C) and column (D), which connection line is suitable for feeding aqueous formate; and

(f) an apparatus (E) suitable for spray granulation, spray drying or melt crystallization, which apparatus is connected on the feed side to the column bottom of column (D).

12. The use of the acid formates prepared as claimed in claims 1 to 8 for preserving and/or acidifying plant and/or animal materials.

13. The use of the acid formates prepared as claimed in claims 1 to 8 for treating biowastes.

14. The use of the acid formates prepared as claimed in claims 1 to 8 as an additive in animal nutrition and/or as growth promoters for animals.

Process and apparatus for preparing acid formates and their use

Abstract

A process for preparing acid formates which comprises

(a) partially hydrolyzing methyl formates with water;

(b) separating off by distillation methyl formate and methanol from the reaction mixture obtained in process stage (a), forming a stream comprising formic acid and water;

(c) converting the stream comprising methyl formate with or without methanol from the process stage (b) by

(i) reaction with a basic compound having a pK_a of the conjugate acid of the appropriate dissociation state of ≥ 3, measured at 25°C in aqueous solution, in the presence of water, and

(ii) removal of the methanol by distillation,

into a stream comprising formate and water; and

(d) combining the stream comprising formic acid and water from the process stage (b) and the stream comprising formate and water from the process stage (c), forming a mixture comprising the acid formate and water,

an apparatus for their preparation and their use.